EFFECTIVE THERMAL CONDUCTIVITY OF EPOXY MATRIX COMPOSITES FILLED WITH RED MUD POWDER

A Project Report Submitted in Partial Fulfillment of the Requirements for the Degree of

B. Tech.

(Mechanical Engineering)

By

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Roll No. 109ME0405

Department of Mechanical Engineering

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

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C E R T I F I C A T E

This is to certify that the work in this thesis entitled ***Effective Thermal Conductivity of Epoxy Matrix Composites Filled With Red mud Powder*** by **Shubhashish Pradhan,** has been carried out under my supervision in partial Fulfillment of the requirements for the degree of **Bachelor of Technology** in *Mechanical Engineering* during session 2012 - 2013 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

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ABSTRACT

Particulate filled polymer composites with enhanced thermo-physical properties are highly in demand in electronic industry and economically viable and environmentally acceptable solutions for industrial waste like red mud are always encouraged. This project presents a numerical, experimental and analytical investigation on the thermal conductivity enhancement of Red mud filled epoxy composites. Guarded heat flow meter test method is used to measure the thermal conductivity of Red mud powder filled epoxy composites using an instrument Unitherm^{TM}Model 2022 according to ASTM-E1530. In the numerical study, the finite-element package ANSYS is implemented to calculate the effective conductivity of the composites. Three-dimensional sphere-in-cube lattice models are used to simulate the microstructure of particle filled composite materials for various filler size and concentration. This study shows that the incorporation of red mud particulates results in the improvement of thermal conductivity of epoxy resin. The experimental conductivity values of composites are compared with the numerically values and it is found that the values obtained using finite element method (FEM) are in proper agreement with the experimental values.

Key Words*: Polymer Composite, Epoxy resin, Red mud, thermal Conductivity, FEM*

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Chapter 1

Introduction

Chapter 1

1. INTRODUCTION

1.1Composite Materials:

Composites Materials are combinations of two materials i.e. the reinforcing phase, which is in the form of fiber sheets or particles and are embedded in the other material called the matrix phase. The matrix phase can be thought of as glue that holds the reinforcing phase. The primary functions of this matrix are to transfer stresses between the reinforcing fibers or particles and to protect them from mechanical and environmental damage whereas the presence of fibers or particles in a composite improves its mechanical properties such as strength, stiffness etc. A composite is therefore a synergistic combination of two or more micro-constituents that are distinct at macroscopic or microscopic level within the finished structure. Our main objective is to take advantage of the superior properties of both materials without compromising the adverse effects. Low density, high strength-to-weight ratio and high tensile strength at higher temperatures, dielectric properties and toughness are the useful properties for which composites have been in demand for various applications. Composites are generally anisotropic materials where the reinforcing materials have low densities with high strength while the matrix is usually a ductile or tough material. If the composite is fabricated correctly depending on their design orientation, volume fraction and other physical properties of constituent materials it provides desirable properties which are not available in any single traditional material.

1.2Types of Composite Materials:

On the basis of the type of matrix material composites are classified into 3 groups.
They are:

a) Metal Matrix Composites (MMC)

b) Ceramic Matrix Composites (CMC)

c) Polymer Matrix Composites (PMC)

a) **Metal Matrix Composites:**

As the name suggests these composites have matrix constituent of metal, while the other can be another metal or a ceramic or an organic compound. These Composites have many superior properties over monolithic metals like higher specific modulus, higher strength, superior properties at elevated temperatures and lower coefficient of thermal expansion. These attributes of metal matrix composites are the main cause for its wide range of applications i.e. combustion chamber nozzle , housings, cables, heat exchangers, high performance sport car, structural members etc.

b) Ceramic matrix Composites:

Ceramic matrix composites are a sub group of composite materials which consists of ceramic fibers embedded into ceramic matrix. Toughness is the main objective behind these ceramic matrix composites. Consequently there is improvement in the strength and stiffness of the composites. These types of composites are used in fields requiring reliability at high temperature and resistance to corrosion and wear. Its major applications include components of gas turbine, disk brake, sliding bearing etc.

c) Polymer Matrix Composites:

These are the most commonly used matrix material due to its low cost and easy fabrication method but its mechanical properties are inadequate for many structural application. Though their strength and stiffness are less than that of metal and ceramic matrix composites, these difficulties are solved by reinforcing other materials with polymers like ceramic. Further the processing of polymer matrix composites do not require high pressure and high temperature which results in easy fabrication process. These characters encourage the use of these types of composites in structural applications. The elastic modulus of these composites is greater than that of the neat polymer but is not as brittle as ceramics.

.1.3 Classification of polymer composites:

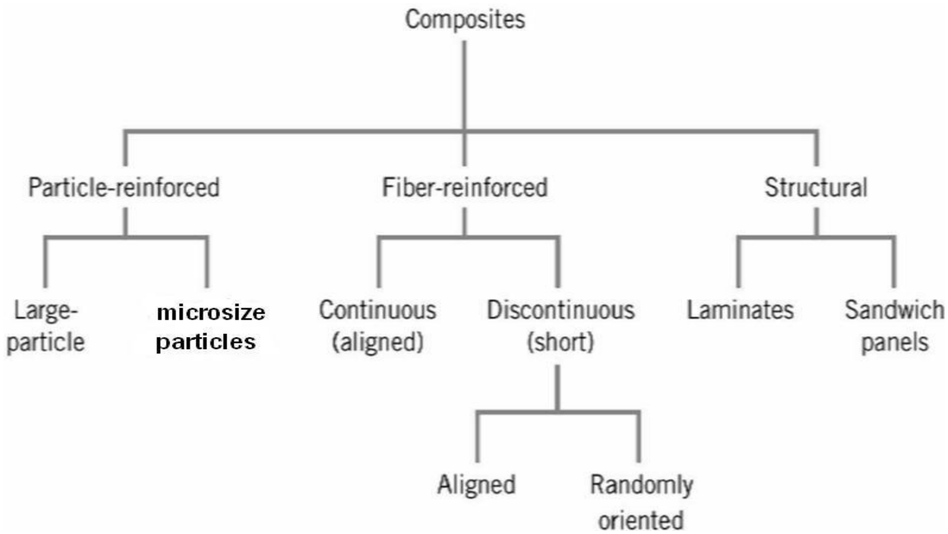

Fig. 1.1: Classification of composites based on reinforcement type

On the basis of the type of reinforcing material Polymer composites can be classified into following three groups. They are:

(a) Fiber reinforced polymer (FRP)

(b) Particle reinforced polymer (PRP)

(c) Hybrid Composites/Structural Composites

1.4 Red Mud:

Red mud is the insoluble product generated after the digestion of bauxite with alkali sodium hydroxide at high temperature and pressure to produce alumina. This process of extraction of alumina is known as Bayer's process. The waste product derives its color and name from its iron oxide content. Red mud is a mixture of compounds originally present in the parent mineral, bauxite and of compounds formed during the Bayer process.

Enormous quantity of red mud is generated worldwide every year posing a very serious and alarming environmental problem. As during the extraction process the bauxite is treated with sodium hydroxide, the red mud possesses high caustic properties (pH 10.5-12.5).

The environmental concern arises due to two aspects: very large quantity of the red mud which results high land consumption and its causticity. The general trend of today for the industrial wastes or by-products is to look for economically viable and environmentally acceptable solution to eliminate cost of disposal and avoid soil and water contamination. Though methods have been developed for maximum recovery of soda and alumina from red mud, other metals can be recovered economically by further investigations to reduce high reaction temperatures required. It can be used as a constructional/building material in formation of bricks, light weight aggregates, further in the cement industry as cements and in concrete industry as well. Bauxite residues can be effective for soil remediation and as a clay material. It can also be as an useful additive to cements, mortars, construction of dykes and as refractory product. In environmental purpose, it can be suitably utilized as adsorbent for cleaning of industrial gases and for waste water treatment. Red mud has it's applications in paints and pigments also.

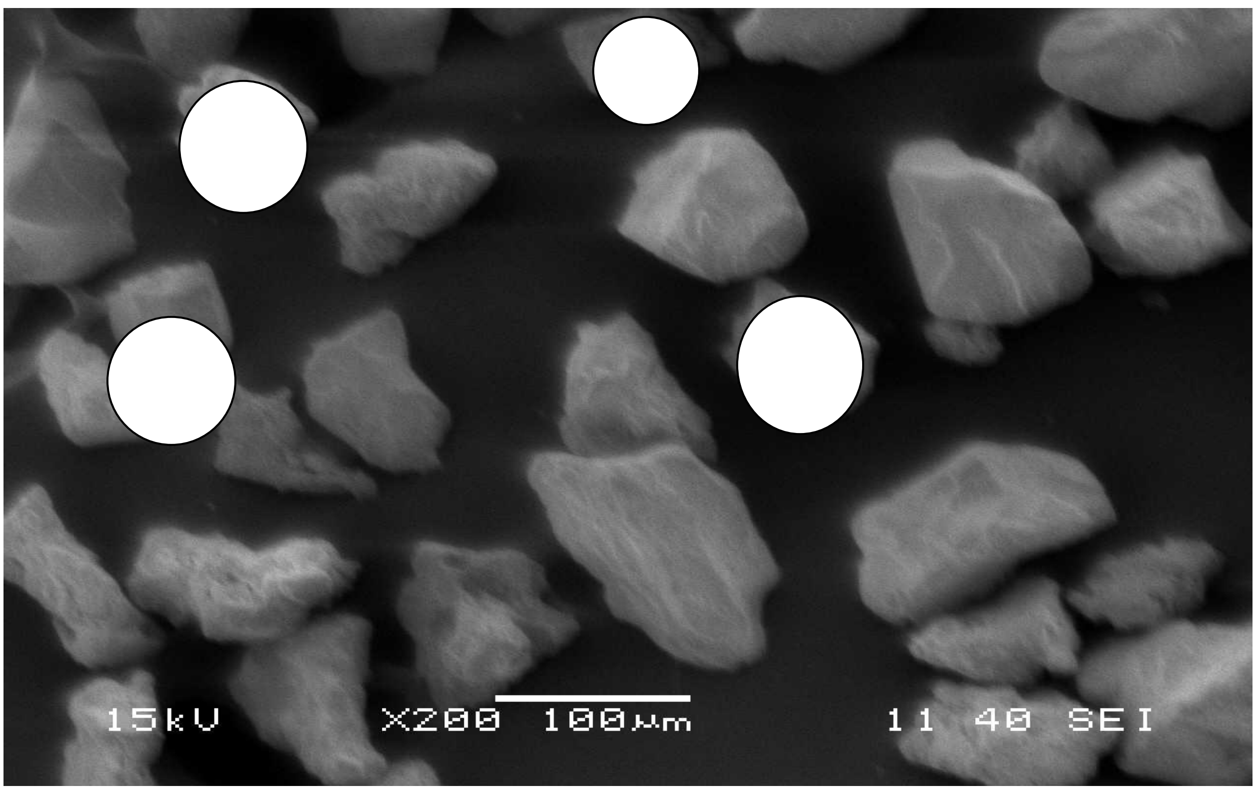

Fig 1.2 SEM Micrograph of spherical red mud particles

Chapter 2

Literature Review

Chapter 2

2. LITERATURE REVIEW

This chapter deals with the background information to be considered in this thesis and focuses on the relevance of the present study. This treatise embraces some related aspects of particle filled polymer composites with special reference to their thermal conductivity characteristics and industrial wastes. This topic includes:

- Particle reinforced polymer composite
- Industrial waste like red mud in the field of composite

2.1 Particle reinforced polymer composite:

polymer matrix reinforced with metal particles have significant industrial applications like heaters, electrodes [1], these composites have thermal durability at high temperature [2] etc. Currently, there has been reduction in the particle size and many studies have researched on the effect of single-particle size on various mechanical properties. The shape, size, volume fraction, and specific surface area of such filler particles have significant effect on mechanical properties of the composites[3]. Research work in the field of thermal conductivity of the polymer has been dominated by Hansen and Ho [4], Peng et. al [5], Choy and Young [6], Tavman [7]. Heat transfer capacity increases in the direction of orientation where as there is slight decrease in the direction perpendicular to the orientation. Griesinger et. al [8] reported a significant increment in the thermal conductivity of low-density poly-ethylene (LDPE) 0.35 W/m-K for an isotropic sample, to 50 W/m-K for a sample with an orientation ratio of 50. Weidenfeller et al. [9] noticed the effect of the interconnectivity of the filler particles on the thermal conductivity of the composites.

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They observed a significant improvement in the thermal conductivity of the PP matrix from 0.27 up to 2.5W/m-K with 30 vol% talc, while same volume fraction of copper particles resulted a thermal conductivity of 1.25 W/m-K despite the fact that copper particles have a thermal conductivity approximately 40 times that of talc particles. Tekce et. al [10] observed the strong influence of the shape of filler particles on the effective thermal conductivity of the composite. Patnaik et. al noticed the existence of a correlation between wear resistance and thermal conductivity of particulate filled composites [11]. [12]Y.P. Mamunya et. Al studied the electrical and thermal conductivity of systems based on epoxy resin (ER) and poly(vinyl chloride) (PVC) filled with metal powders. Copper and nickel powders having different particle shapes were taken as fillers. The preparation conditions allowed the formation of a random distribution of metallic particles in the polymer matrix volume for the systems Epoxy–Cu, Epoxy–Ni, PVC–Cu and to create ordered shell structure in the PVC–Ni system. Particle shape and type of spatial distribution greatly influence the thermal conductivity. N.M. Sofian et. Al [13] investigated experimentally the thermal properties such as thermal conductivity, thermal diffusivity, specific heat of metal (copper, zinc, iron, and bronze) powder-filled high-density polyethylene composites in the range of filler content 0–24% by volume. Experimental results show a region of low particle content, 0–16% by volume, where the particles are distributed homogeneously in the polymer matrix and do not interact with each other. At higher particle content, the filler tends to form agglomerates and conductive chains resulting in a rapid increase in thermal conductivity

2.2 Industrial waste like red mud in the field of composites:

Though Waste management and recycling processes have been adopted to neutralize the hazardous effect of industrial waste still it has been a major environment concern. Apart from the neutralization of industrial waste much attention has been devoted for economic viable and environmentally acceptable solutions. Red mud, an industrial waste generated during the extraction of alumina from bauxite has been a huge environment concern due it's large production which causes disposal problem and it's alkalinity which has augmented the environment concern.

It was the waste removal technique that had been the only hope before various inventions regarding the reusability of these industrial wastes. Red mud for instance can be used in aluminum metal matrix composites for it's wear resistance applications, along with other tribological properties [16,17]. Then S.C. Mishra et al. analysed the the tribological behavior of red mud composite coatings. Room temperature solid particle erosion trials are carried out using a compressed air blasting type rig under impact angles of 30º, 60º and 90º.The investigation used an erosion apparatus of Sand Blast type .The test is conducted as per ASTMG76 standards. It is analyzed that initially the cumulative coating mass loss increases rapidly and later on becomes almost sluggish. During a transient regime in the erosion process the incremental rate of erosion reduces monotonically down to a steady state erosion rate[18]. S.C. Mishra et al. suggested that red mud can be used to deposit ceramic coatings which may be useful for tribological applications. Similarly synergistic effect of copper slag and red mud improve physical and mechanical properties of bamboo fiber reinforced composites [19]. Again Biswas and Satapathy [20] observed that copper slag can be used as filler material for the preparation of composite materials to be used as abrasive and cutting tools. Alumina particulates filled copper matrix composite materials are strengthened by pressure molding and sintering.

The compactness of composites increased with the sintering temperature and increase in pressure, and decreased with the alumina content increasing. The hardness of composite materials increased with the increase of sintering temperature, pressure and alumina particulates [21]. Another abundantly available industrial waste is blast furnace slag which along with limestone powder forms composite which has high compressive strength and improved tensile strain capacity[22]. Cheng and Chiu [23] researched the usage of granulated blast furnace slag as a filler material in making of geo-polymers.

Chapter 3

Materials and Methods

Chapter 3

3. MATERIALS AND METHODS

This chapter showcases the materials and methods used for the processing of the particulate filled polymer composites. It presents the details of the characterization and thermal conductivity tests which the composite samples are subjected to. Further the numerical methodology for the determination of the effective thermal conductivity based on finite element method is also presented in this chapter.

3.1 MATERIALS

3.1.1 Matrix Material:

Epoxy LY 556 resin, chemically belonging to the „epoxide" family is used as the matrix material in this present research procured from Ciba-Geigy India Ltd. Its common name is Bisphenol-A-Diglycidyl-Ether. The low temperature curing epoxy resin (Araldite LY 556) and the corresponding hardener (HY 951) are mixed in a ratio of 10:1 by weight as recommended. Epoxy is chosen primarily because it happens to the most commonly used polymer and because of its insulating nature (low value of thermal conductivity, about 0.363 W/m-K) and low density (1.1 gm/cc).

3.1.2 Filler Material (Red mud powder):

Red mud is the insoluble product generated after the digestion of bauxite with alkali sodium hydroxide at high temperature and pressure to produce alumina. This process of extraction of alumina is known as Bayer's process. It has wide range of application starting from in the field of building (clay material, cements, ceramics, fired and non-fired building materials, concrete industry), pollution control (treatment of waste water and polluted waste gases), metal recovery (iron, titanium, aluminum, alkali), coagulant, adsorbent, catalyst and in soil remediation. Its thermal conductivity and density values are 12 W/m-K and 3.3 gm/cc respectively.

3.2 Composite Fabrication:

The low temperature curing epoxy resin (LY 556) and corresponding hardener (HY951) supplied by Ciba Geigy India Ltd. are mixed in a ratio of 10:1 by weight as recommended. Red mud powder (density 3.3 gm/cc) with average size 100 μm are reinforced in epoxy resin (density 1.1 gm/cc) to prepare the composites. The dough (epoxy filled with Red mud powder) is then slowly poured into the moulds, coated beforehand with wax and uniform thin film of silicone-releasing agent for its excellent releasing characteristics. The composites are cast by conventional hand-lay-up technique so as to get disc-shaped specimens. Composites of six different compositions (with 1.4, 3.35, 5.23, 7.85, 9.4 and 11.3 vol % of red mud respectively) are made. The castings are left for 24 hours to cure at room temperature and then samples are released by breaking the moulds.

Samples	Composition (for red mud filled epoxy)
1	Epoxy + 1.4 vol% (1.05 wt %) Filler
2	Epoxy + 3.35 vol% (2.51 wt %) Filler
3	Epoxy + 5.23vol% (3.92 wt %) Filler
4	Epoxy + 7.85 vol% (5.88wt %) Filler
5	Epoxy + 9.4 vol % (7.05 wt %) Filler
6	Epoxy + 11.3 vol% (8.48 wt%) Filler

Table 3.1: List of particulate filled composites fabricated by hand-lay-up technique

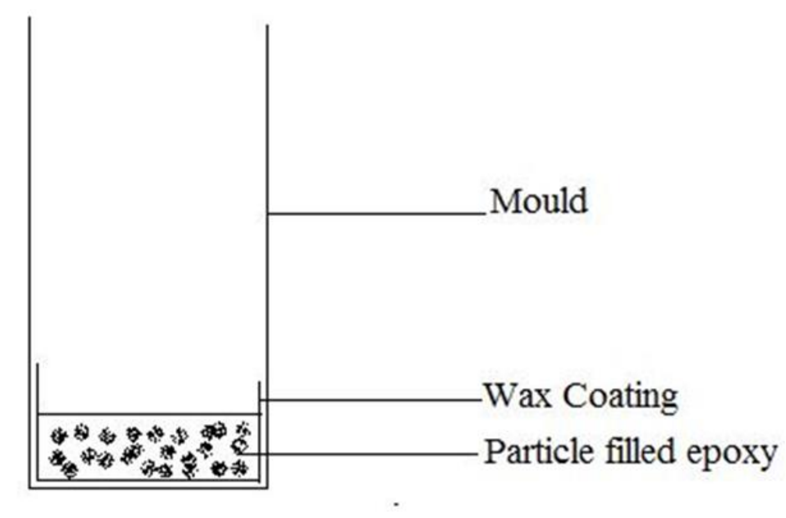

Fig. 3.1 Preparation of particulate filled composites by hand-lay-up technique

3.3 THERMAL CHARACTERIZATION

3.3.1 Experimental Determination of Thermal Conductivity:

Unitherm™ Model 2022 is a thermal conductivity measuring instrument for a variety of materials that includes polymers, ceramics, composites, some metals and other materials of thermal conductivity from low to medium range. It requires a small test sample only. Non-solids like thin films can also be tested accurately employing a multi-layer technique.

The tests are according to **ASTM E-1530** Standard.

3.3.2 Operating principle of UnithermTM 2022:

A sample is held under a uniform compressive load between two polished surfaces which regulates the temperature of the two surfaces. The direction of heat flow is from the top surface, through the sample, to the bottom Surface which results an axial temperature gradient in the stack. After thermal equilibrium is reached, the temperature difference across the sample (temperature difference between upper and lower surface) is measured along with the output from the heat flow transducer These values and the sample thickness are the input values used to calculate the thermal conductivity. With the help of temperature sensors on either side of the sample The drop in temperature through the sample is measured.

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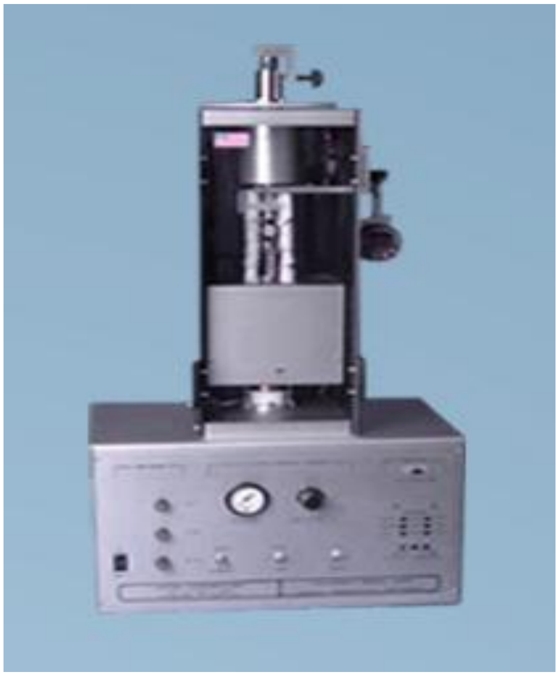

Fig. 3.2 Determination of Thermal Conductivity Using Unitherm™ Model 2022

Thermal conductivity of a material is defined as the rate of heat flow with in a body for a specific temperature difference. For one-dimensional heat flow by conduction the formula can be given as equation 3.1:

$$Q = \frac{KA(T1-T2)}{x} \qquad (3.1)$$

Where Q is the heat flux (W)

K is the thermal conductivity of the body(W/m-K)

A is the cross sectional area (m^2)

T_1-T_2 is the difference in temperature across the body(K)

x is the thickness of the sample (m).

The thermal resistance of a sample can be stated as:

$$R = \frac{T1-T2}{(Q/A)} \qquad (3.2)$$

Where, R is the resistance of the sample between surfaces (upper and lower)(m^2-K/W). From Equations 3.1 and 3.2 we can derive that

$$K = \frac{x}{R} \qquad (3.3)$$

In Unitherm 2022, the heat flux transducer measures the Q value and the temperature difference can be obtained between the upper and lower plate. Giving the input value of thickness of the sample and the known cross sectional area, the effective thermal conductivity of the composite samples can be calculated using Equation 3.3.

3.4 Numerical Analysis: Concept of Finite Element Method (FEM) and ANSYS:

The Finite Element Method (FEM) was introduced by Turner et al. [24] in 1956, which is a powerful computational technique for approximate solutions for a variety of engineering problems with complex domains subjected to general boundary conditions. FEM has become a crucial step in the modeling of a physical phenomenon in various engineering fields. As the field variables vary from point to point, it results in an infinite number of solutions with in the domain.

The basic concept of FEM relies on the decomposition of the domain into a finite number of subdomains (the sample into finite number of elements) for which the systematic approximate solution is constructed by applying either variational or weighted residual methods. FEM reduces the problem into a finite number of unknowns by dividing the domain into elements and expresses the unknown field variable in the form of assumed approximating functions within each element. These functions are also called interpolation functions. These functions define the values of the field variables at specific points called nodes. Nodes connect adjacent elements. This method has the ability to discretize the irregular domains with finite elements for which it is a valuable and practical analysis tool for the solution of boundary and eigen value problems arising in various engineering fields.

3.4.1 Basic Steps in FEM:

The very first step is to convert the governing differential equation into an integral form. The two techniques to achieve this are :

(i) Variational Technique

(ii) Weighted Residual Technique.

In variational technique, the integral form corresponding to the given differential equation is obtained by using calculus of variation. The solution of the problem can be obtained by the minimization of the integral. In weighted residual technique, the weighted integrals of the governing differential equation are constructed where the weight functions are known and arbitrary except that they satisfy boundary conditions. Often this integral form is modified using the divergence theorem To reduce the continuity requirement of the solution. Then solution is obtained by setting the integral to zero.

3.4.2 Advantages of the finite element method over other numerical methods are as follows:

- Any irregular-shaped domain or any type of boundary condition can be analyzed using this method.
- Analysis of domains consisting more than one material can be easily done.
- However the accuracy of the solution can be improved by implementing refinement of the mesh or by choosing higher degree polynomials.

3.5 Analytical Model:

3.5.1 Sphere in cube

The effective thermal conductivity of the cube in cube model has been derived from the existing sphere in cube model proposed by J.Z. Liang and G.S.Liu. The final expression of this model is:-

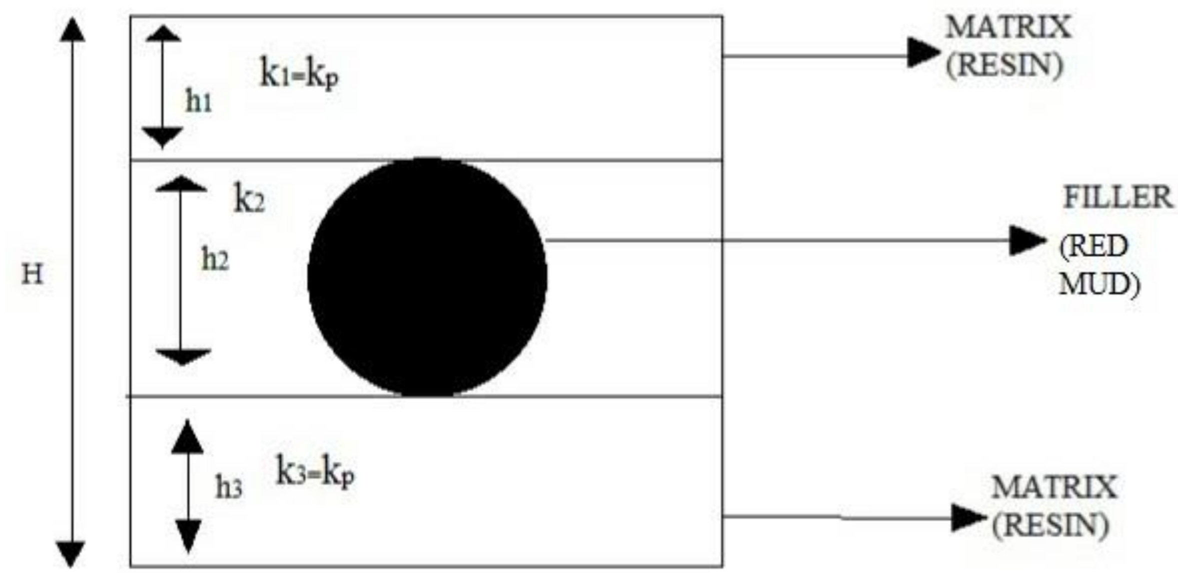

Fig 3.3 sphere in cube model

$$k_{eff} = \frac{1}{\frac{1}{k_p} - \frac{1}{k_p}\left(\frac{6\Phi}{\pi}\right)^{1/3} + \frac{4}{k_p\left(\frac{4\pi}{3\Phi}\right)^{2/3} + (K_f - K_p)\left(\frac{16\pi^2}{9}\right)^{1/3}}}$$

Where k_p= Thermal conductivity of epoxy polymer

k_f=Thermal conductivity of filler material

Φ= volume fraction of filler material

3.5.2 Cube in cube model:

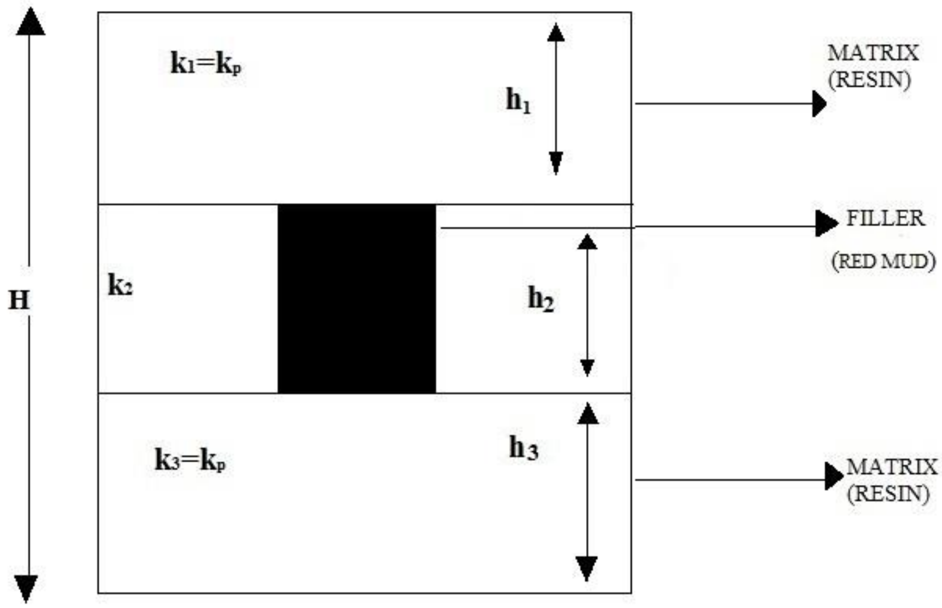

Fig 3.4 Cube in cube

Here $k_{eff} = \dfrac{m}{\frac{(m-1)}{k_p} + \frac{m^2}{k_p(m^2-1)+K_f}}$ where $m=\left(\frac{1}{\phi}\right)^{\frac{1}{3}}$

Where k_p= Thermal conductivity of epoxy polymer

k_f=Thermal conductivity of filler material

ϕ= volume fraction of filler material

Chapter 4
Results and Discussion

Chapter 4

4. RESULTS AND DISCUSSION

4.1 Description of the problem:

For functional design and application of composite materials the most important parameter is the effective properties of composite. The important factors that regulate the effective properties is the microstructure of the composite and it can be controlled to an appreciable level. Microstructure of composite includes the size, shape, spatial distribution and orientation of the reinforcing inclusion in the matrix. Although most inclusions have random distributions, periodic structure can provide the details of the effect of microstructure on the effective properties can be. System with periodic structures is easy to analyze because of the high degree of symmetry.

Thermal analysis is carried out for the conductive heat transfer through the composite body using the finite-element program ANSYS. For the thermal analysis purpose, three-dimensional physical models with spheres-in-a-cube and cube in cube lattice array have been used to simulate the microstructure of composite materials for six different filler concentrations. In numerical method the effective thermal conductivities of these red mud powder filled epoxy composites up to about 11.3% filler content is determined by using ANSYS.

4.1.1 Assumptions:

For the ideal case thermal analysis it will be assumed that:

1. The composites are considered to be macroscopically homogeneous.
2. Both the matrix and filler are homogeneous and isotropic.
3. The thermal contact resistance between the matrix and the filler is assumed to be negligible.
4. Voids are not present in the composite lamina.
5. The problem considered is based on 3D physical model.
6. The filler are arranged in a square periodic array/uniformly distributed in matrix.

4.2. Numerical Analysis

In the numerical analysis of the problem, the temperatures at the nodes of the surface ABCD is prescribed as T1 (=100^{0}C) and the convective heat transfer coefficient is assumed to be 2.5 W/m^2-K at ambient temperature of 27°C. The heat flow direction and the boundary conditions are shown in Fig. 4.1(heat flow from face ABCD to EFGH). The other surfaces are all assumed to be adiabatic. The unknown temperatures at the nodes in the interior region and on the adiabatic boundaries are obtained with the help of finite-element program package ANSYS.

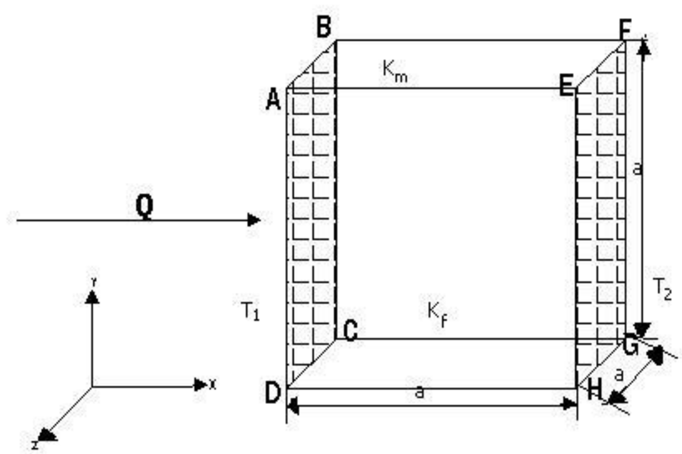

Fig.4. 1 Boundary conditions

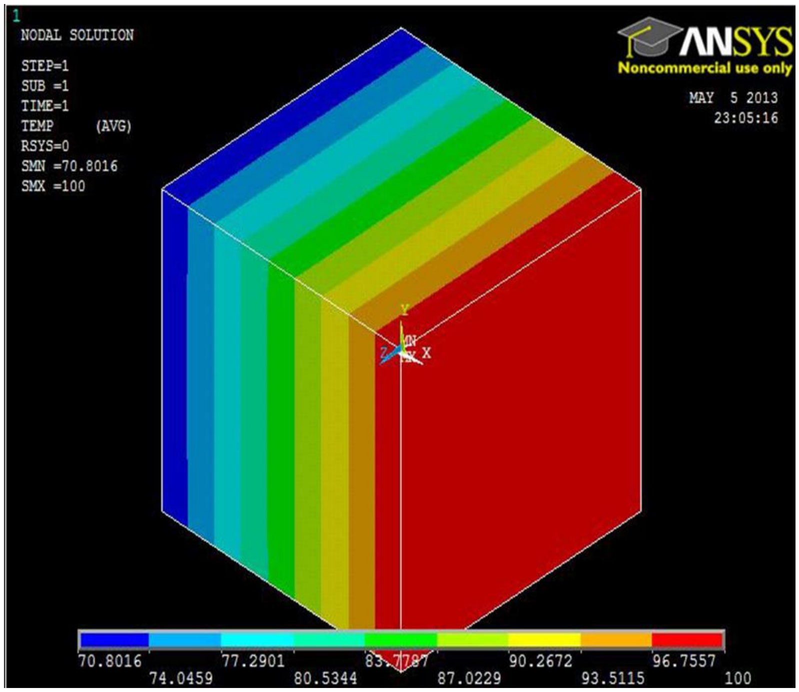

Fig 4.2.1 Temperature profile for red mud-epoxy composite with filler concentration of 1.4%

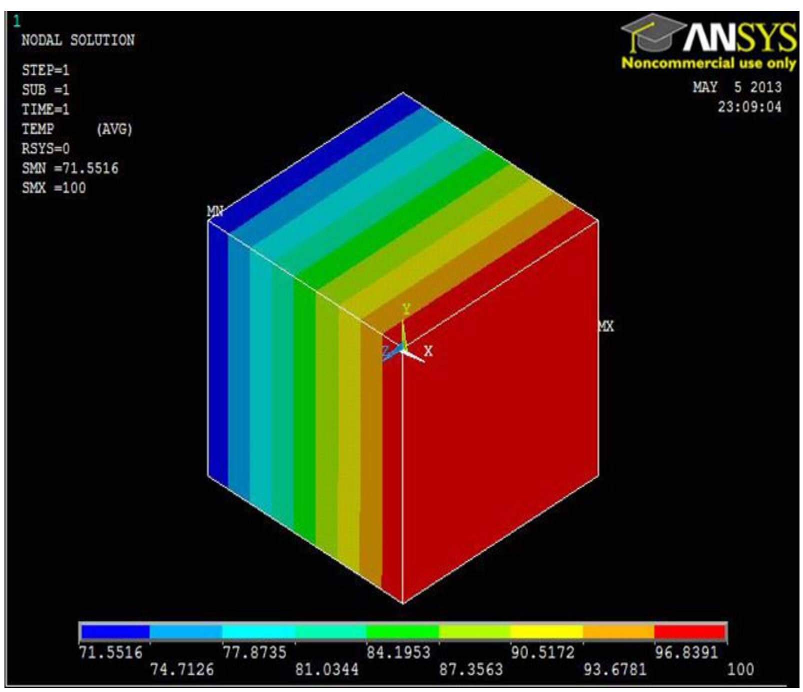

Fig 4.2.2 Temperature profile for red mud-epoxy composite with filler concentration of 3.35%

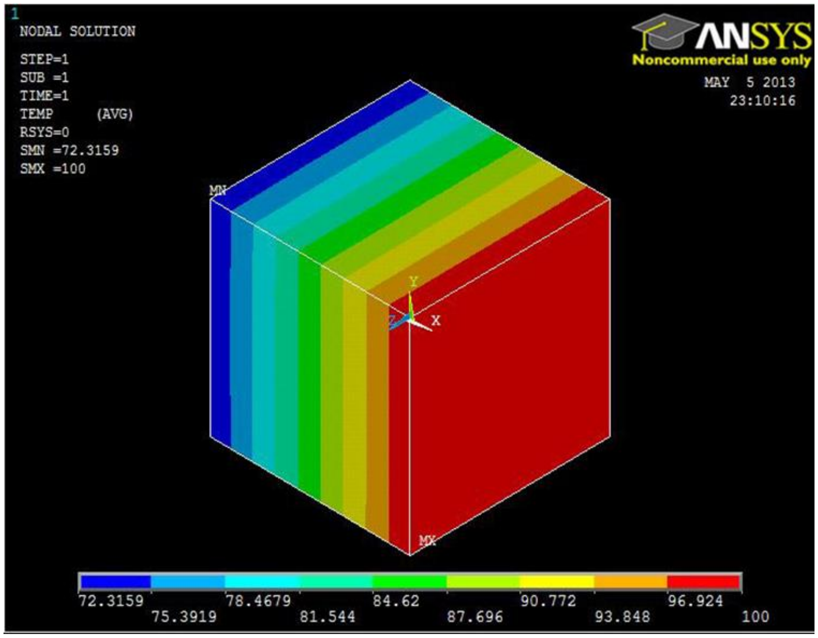

Fig 4.2.3 Temperature profile for red mud-epoxy composite with filler concentration of 5.25%

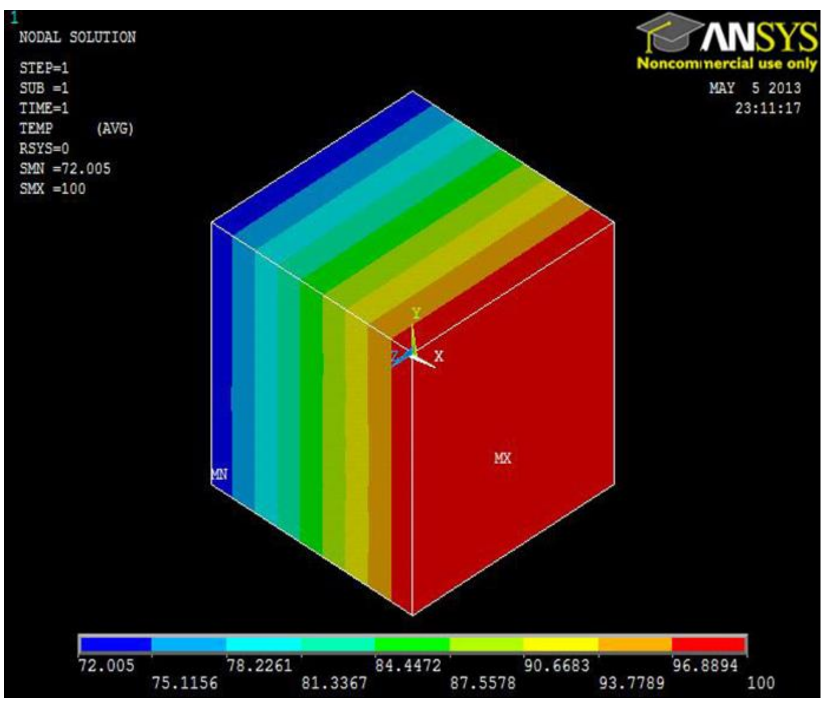

Fig 4.2.4 Temperature profile for red mud- epoxy composite with filler concentration of 7.85%

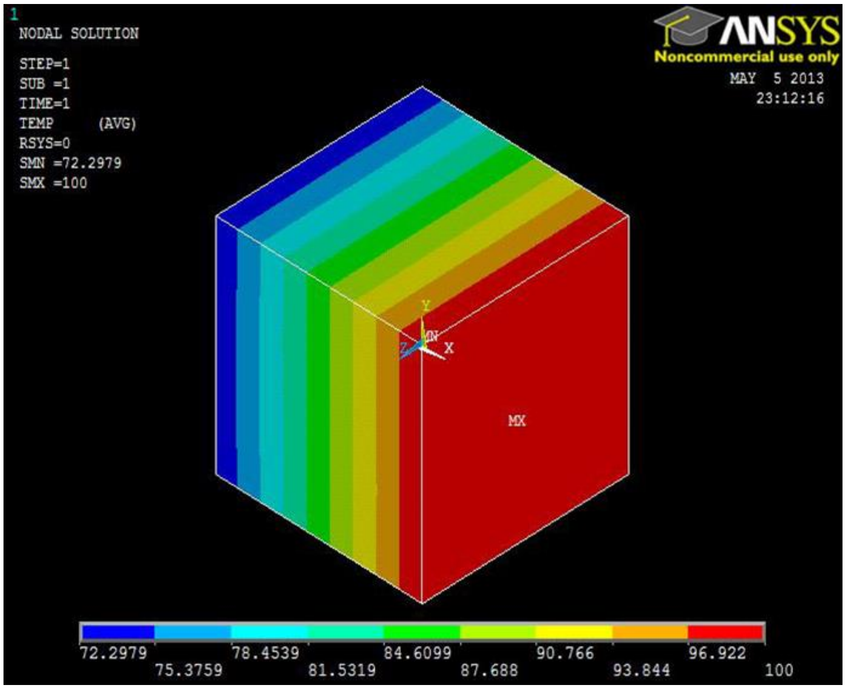

Fig 4.2.5 Temperature profile for red mud-epoxy composite with filler concentration of 9.4%

Table 4.1 Thermal conductivity for composites obtained from FEM, Analytical model and Experiment

Sample No	Red Mud Content Vol %	Effective Thermal Conductivity		
		FEM Simulated value (sphere in cube)	Experimental Measured	Analytical Model
1	0	0.363	0.363	0.363
2	1.4	0.38	0.376	0.5
3	3.35	0.4	0.393	0.57
4	5.23	0.42	0.406	0.63
5	7.85	0.44	0.429	0.71
6	9.4	0.45	0.438	0.76
7	11.3	0.46	0.449	0.81

Table 4.2 Percentage errors associated with the FEM simulated values with respect to the measured values (for red mud filled epoxy composites)

Composite Sample	Red Mud Content (Vol. %)	Percentage errors associated with FEM results w.r.t. the experimental value (%)
1	1.4	1.06
2	3.35	1.78
3	5.23	3.44
4	7.85	2.56
5	9.4	2.73
6	11.3	2.44

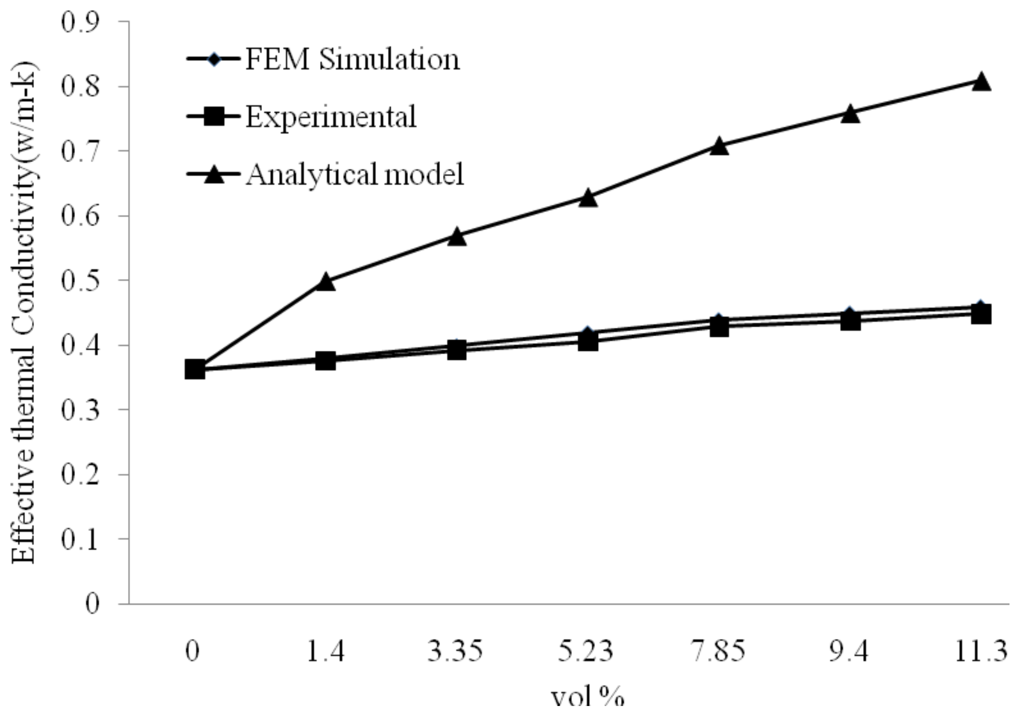

Fig 4.3 Comparison between Experimental values, Analytical values and FEM Analysis

Thermal conductivities of epoxy composites filled with red mud powder particles from 1.4% to 11.4% by volume are numerically estimated by using the finite element package ANSYS (spheres-in-cube model) and the numerical results are compared with the experimental results of the thermal conductivity of samples and also with the analytical model. The temperature profiles obtained from FEM analysis for the composites with filler concentrations of 1.4, 3.35, 5.23, 7.85, 9.4 and 11.3 vol% are presented in Figures 4.2.1 - 4.2.5 respectively

This study shows that finite element method can be suitably employed to determine effective thermal conductivity of the polymer composite with different percentage of filler. The value of equivalent thermal conductivity obtained for various composite models using finite element package ANSYS are in proper agreement with the experimental values calculated using Unitherm™ Model 2022 for a wide range of filler contents from about 1.4 vol% to 11.3 vol%. Incorporation of red mud results in enhancement of thermal conductivity of epoxy resin. With addition of 1.4 % and 11.3% of red mud the thermal conductivity increases by about 6.61%and 29.75 % respectively when compared with neat epoxy resin.

Chapter 5
Conclusions

Chapter 5

5. CONCLUSIONS

- Successful fabrication of red mud powder filled epoxy composites is carried out by Hand-lay-up technique.

- Despite of being an industrial waste red mud can be used as a filler material in the epoxy polymer matrix.

- Finite Element Method (FEM) can be suitably employed to determine the effective thermal conductivity (**keff**) of these particulate filled polymer composites for different volume fractions of red mud.

- The values of the effective thermal conductivity (**keff**) obtained for various composite models from FEM are in proper agreement with the experimental values for a wide range of filler content from 1.4 vol% to 11.3 vol%.

- Inclusion of red mud particles results in the improvement of thermal conductivity of epoxy resin. With addition of 11.3 vol. % of red mud, the thermal conductivity improves by about 29.75 % with respect to neat epoxy resin.

- These new class of red mud particle filled epoxy composites can be suitably applied for applications such as electronic packages, encapsulations, heat sink, thermal interface material.

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